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<p>(21) International Application Number: PCT/US97/20620</p> <p>(22) International Filing Date: 13 November 1997 (13.11.97)</p> <p>(30) Priority Data: 08/748,714 14 November 1996 (14.11.96) US</p> <p>(63) Related by Continuation (CON) or Continuation-in-Part (CIP) to Earlier Application US 08/748,714 (CON) Filed on 14 November 1996 (14.11.96)</p> <p>(71) Applicant (for all designated States except US): THE GILLETTE COMPANY [US/US]; Prudential Tower Building, Boston, MA 02199-8004 (US).</p> <p>(72) Inventor; and (75) Inventor/Applicant (for US only): PUNO-D'ANTONIO, Maria [PH/US]; 348 Forest Avenue, Cohasset, MA 02025 (US).</p> <p>(74) Agents: HANDELMAN, Joseph, H.; Ladas &amp; Parry, 26 West 61st Street, New York, NY 10023 (US) et al.</p>		<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).</p> <p><b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
<p>(54) Title: <b>CONDITIONING SHAMPOO</b></p> <p>(57) Abstract</p> <p>Conditioning shampoo compositions are provided. Preferred conditioning shampoo compositions include an insoluble silicone, a stabilizing agent including a glyceryl ester and a smectite clay, an amino-functional silicone derivative, a pearl pigment, and water.</p>		

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CONDITIONING SHAMPOO

The invention relates to conditioning shampoos.

5 Shampooing has long been used to remove dirt and excess oil and sebum from human hair. While shampoo compositions are generally effective in cleansing the hair, shampooing tends to leave the hair in a tangled and unmanageable state. Accordingly, conditioners have been developed to prevent this occurrence and to add body, shine, and other desired properties to the hair. Such conditioners are often applied  
10 to the hair in a separate step, after the shampoo has been rinsed out, allowed to remain on the hair for a short period, and rinsed out. Some shampoo users view applying a conditioner after shampooing, waiting the recommended period and having to rinse again, as an inconvenience. As a result, combination "conditioning shampoos" have been developed to allow the hair to be shampooed and conditioned in a single application step.

15 The present invention features a conditioning shampoo that provides good cleaning, conditioning, and rinsability. In addition, the shampoo imparts increased volume and body to the hair, does not cause significant greasy build-up even with frequent use, and generally leaves hair feeling soft and shiny. The shampoo also has an aesthetic pearlescent appearance, and long shelf stability.

20 In one aspect, the invention features a conditioning shampoo including a cleansing agent, an insoluble silicone conditioning agent, a stabilizing agent comprising a glyceryl ester and a smectite clay, and water.

Preferably, the cleansing agent includes ammonium laureth sulfate, ammonium lauryl sulfate, sodium lauryl sulfate, lauramide DEA, cocamidopropyl betaine, and ammonium xylene sulfonate. More preferably, the shampoo includes these cleansing  
25 agents in the following amounts, by weight percent based on the total weight of the active ingredients in the shampoo (referred to hereinbelow as "% Active"): from about 9 to 10% ammonium laureth sulfate, from about 3 to 4% ammonium lauryl sulfate, from about 3.5 to 4.5% sodium lauryl sulfate, from about 2 to 3% lauramide DEA, from about 1 to 2%  
30 cocamidopropyl betaine, and from about 0.25 to 0.75% ammonium xylene sulfonate. Preferred insoluble silicone conditioning agents include a mixture of a non-volatile polyalkyl siloxane and an amino-functional silicone derivative. Preferably the polyalkyl siloxane is dimethicone and the amino-functional silicone derivative is

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5 trimethylsilylamodimethicone. Preferred glyceryl esters include esters of diacetyl tartaric acid glyceryl stearate. A particularly preferred glyceryl ester is the emulsifier having the CTFA/INCI name "DATEM". Preferably, the smectite clay is a magnesium aluminum silicate, and the DATEM and magnesium aluminum silicate are provided in a weight ratio of from about 3:1 to 10:1. Preferred shampoos further include a pearling pigment. In  
10 these shampoos preferably the pearling pigment and the smectite clay are provided in a weight ratio of from about 1:3 to 1:8. In preferred shampoos, the stabilizing agent is provided in an amount sufficient to enable the shampoo to exhibit no visible phase separation after at least 10 temperature cycles wherein during each cycle the shampoo is held for 16 hours at 45°C, followed by 24 hours at 5°C, followed by 8 hours at 25°C. In  
15 particularly preferred shampoos, the shampoo also exhibits no visible phase separation after at least 10 freeze/thaw cycles.

In another aspect, the invention features a conditioning shampoo including a cleansing agent comprising, based on the total weight of the shampoo, from about 9 to 10% ammonium laureth sulfate; from about 3 to 4% ammonium lauryl sulfate, from about  
20 3.5 to 4.5% sodium lauryl sulfate, from about 2 to 3% lauramide DEA, from about 1 to 2% cocamidopropyl betaine, and from about 0.25 to 0.75% ammonium xylene sulfonate; an insoluble non-volatile silicone conditioning agent; a stabilizing agent; and water.

In a further aspect, the invention features a conditioning shampoo including a cleansing agent, a conditioning agent, a pearl pigment, a magnesium aluminum silicate,  
25 and water.

The invention also features methods of using these conditioning shampoos to clean and condition hair by applying the shampoo to wet hair and massaging the hair to form a lather, and rinsing the lather from the hair.

In yet another aspect, the invention features methods of making the conditioning shampoos. A preferred method includes the following steps: (a) dispersing  
30 the smectite clay and the pearling pigment in water to form a pigment dispersion; (b) adding one or more cleansing agents to the pigment dispersion; (c) adding the glyceryl ester and the insoluble silicone to the pigment dispersion and mixing until a stable emulsion is formed; and (d) adding the amino-functional silicone to the emulsion.

35 Preferably, step (a) is performed at about 70 to 80°C, and includes predispersing the

5 smectite clay in the water, adding the pigment, and mixing the clay, pigment and water until a desired viscosity is obtained, e.g., for about 40 to 70 minutes.

It is also preferred that step (c) be performed at about 70 to 80°C and include adding the glyceryl ester to the pigment dispersion, mixing until uniform, e.g., for about 15 to 30 minutes, and then adding the insoluble silicone and mixing until emulsified, e.g., for about  
10 40 to 70 minutes. Preferably the emulsion is cooled, e.g., to about 50°C, before the amino-functional silicone derivative is added, and further cooled, e.g., to 35°C, before any other additives are added to the shampoo.

The term "insoluble silicone," as used herein, refers to a silicone that is insoluble in the shampoo composition matrix.

15 The term "stable emulsion," as used herein, refers to an emulsion which will exhibit no visible phase separation after at least one temperature cycle wherein the emulsion is held for 16 hours at 45°C, followed by 24 hours at 5°C, followed by 8 hours at 25°C.

Other features and advantages of the invention will be apparent from the  
20 description of preferred embodiments thereof, taken together with the drawings, and from the claims.

Preferred conditioning shampoo compositions include one or more cleansing agents, an insoluble silicone, an amino-functional silicone derivative, a pearl pigment, a stabilizing agent, and water.

25 Suitable cleansing agents include anionic surfactants such as alkyl sulphates, alkyl ether sulphates, alkaryl sulphonates, alkyl succinates, alkyl sulphosuccinates, N-alkoyl sarcosinates, and alpha-olefin sulfonates. Preferred anionic surfactants include sodium lauryl sulphate, ammonium lauryl sulphate, and ammonium laureth sulphate. Other suitable cleansing agents include amphoteric surfactants, such as  
30 alkyl amine oxides, alkyl betaines, alkyl amidopropyl betaines, alkyl sulphobetaines, alkyl glycinate, and alkyl carboxyglycinates. Preferred amphoteric surfactants include cocamidopropyl betaine. Non-ionic surfactants may also be included, e.g., condensation products of aliphatic primary or secondary linear or branched chain alcohols, or phenols with alkylene oxides, e.g., ethylene oxide, for example mono- or di-alkyl alkanolamides  
35 or alkyl polyglucosides. Preferably, the shampoo includes a mixture of the following



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5       cleansing agents, preferably in the amounts in parentheses, based on the total weight of the  
composition: ammonium laureth sulfate (9-10%), ammonium lauryl sulfate (3-4%),  
sodium lauryl sulfate (3.5-4.5%), lauramide DEA (2-3%), cocamidopropyl betaine  
(1-2%), and ammonium xylene sulfonate (0.25-0.0.75%). This blend has been found to  
enhance lathering and foaming of the shampoo, a property which tends to be compromised  
10       when silicones are added to shampoo formulations. This blend also provides good  
cleaning and rinsability. Preferably, the total amount of the cleansing agents, on a total  
weight basis (not % Active), ranges from about 35 to 65, more preferably 45 to 55, percent  
by weight based on the total weight of the composition. If too much of any cleansing  
agent is included, too much lather may be generated during shampooing, making it  
15       difficult to rinse the hair and stripping the conditioning agents from the hair during  
rinsing; if too little is included, the hair may not be completely cleaned during normal use  
of the shampoo and the shampoo may not generate sufficient lather.

Suitable insoluble silicone include non-volatile polyalkyl siloxanes.  
Preferred polyalkyl siloxanes include those available from General Electric under the  
20       tradename VICASIL, and those available from Dow Corning under the tradename  
DC 200. A particularly preferred polyalkyl siloxane is dimethicone. Preferred siloxanes  
have a viscosity of from about 500 to 100,000 centistokes. Preferably, the insoluble  
silicone is present in an amount of from about 0.10 to 5.0, more preferably 0.50 to 2.0, %  
Active. If too much insoluble silicone is included, the shampoo may be unstable and may  
25       leave hair feeling dirty, with an oily or greasy residue (i.e., the hair may be  
overconditioned); if too little is included, the shampooed hair may be difficult to comb  
and under-conditioned (i.e., the hair may be underconditioned).

Suitable amino-functional silicone derivatives include water-insoluble,  
non-volatile amino-functional siloxanes. A preferred amine-functional silicone derivative  
30       is trimethylsilylamodimethicone, which has a viscosity of from about 50 to 500  
centistokes at 25°C. The amino-functional silicone is preferably provided in the form of a  
water-miscible emulsion, more preferably an emulsion in which the siloxane droplets have  
a particle size of from about 50 to 600 micron. This siloxane is commercially available  
from the General Electric Company as SF1705, or in the form of an emulsion as  
35       SM-21115, or from Dow Corning Chemical Company as Dow Corning Q2-8220, or in the

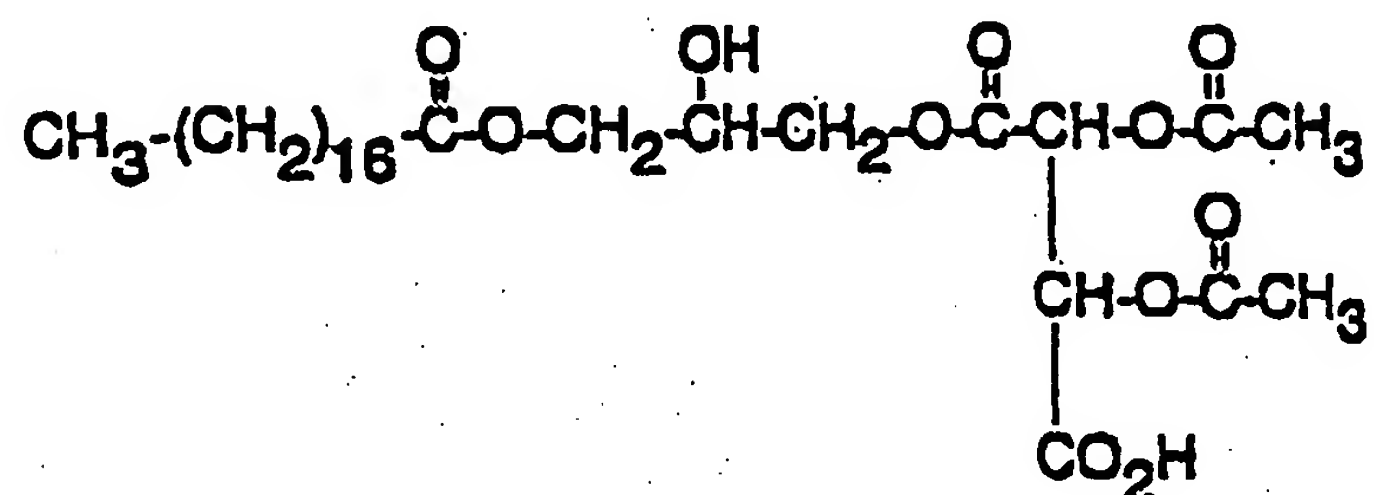


5 form of an emulsion as Dow Corning 929 emulsion or Dow Corning Q2-7224.  
 Preferably, the amino-functional silicone derivative is present in an amount of from about  
 0.01 to 1.0, more preferably 0.15 to 0.5, % Active. If too much is included,  
 overconditioning may result; if too little is included, the hair may be underconditioned.

10 Preferably, the insoluble silicone and amino-functional silicone derivative  
 are provided in a ratio of from about 2:1 to 5:1. It has been found that when these  
 components are provided in a ratio within this range, the shampoo provides good cleaning  
 and conditioning, and leaves the hair with increased body and a clean feel.

The stabilizing agent preferably includes, in combination, an emulsifier for  
 the insoluble silicone and a co-emulsifier/suspending agent that is believed both to  
 15 stabilize the insoluble silicone emulsion and to suspend the pearling pigment.

Suitable emulsifiers are those capable of forming a stable oil-in-water  
 emulsion with the insoluble silicone. Preferred compositions include glyceryl esters, more  
 preferably esters of diacetyl tartaric acid glyceryl stearate, which are believed to function  
 as emulsifiers. A preferred glyceryl ester is commercially available from Goldschmidt  
 20 Chemical Corporation under the tradename AMILAN® GST 40 (the CTFA/INCI name of  
 this compound is "DATEM"). This ester is produced by reacting glycerol, stearic acid  
 and diacetyl tartrate, is an amorphous, high viscosity liquid, and has the following  
 chemical structure:



30

DATEM is strongly substantive to protein surfaces, and thus provides good hair  
 conditioning without build-up, in addition to emulsifying the silicone and stabilizing the  
 35 shampoo.

5 the shampoo will tend to be undesirably high; if too little is included, the emulsion will tend to be unstable.

Suitable coemulsifier/suspending agents are those that are capable of ensuring the stability of the silicone emulsion and maintaining the pearl pigment suspended in the shampoo matrix during the normal shelf life of the shampoo, e.g.,  
10 for 24 months. Preferred shampoos include inorganic clays, preferably refined smectite (montmorillonite) clays characterized by an expanding lattice structure, which are believed to function as suspending agents and co-emulsifiers. A preferred clay is magnesium aluminum silicate (CTFA/INCI name), a complex, colloidal clay. Preferred magnesium aluminum silicates have an approximate average chemical analysis as follows:  
15 55-65% silicon dioxide, 10-15% magnesium oxide, 1% ferric oxide, 2-3% calcium oxide, 2-3% sodium oxide, 1-2% potassium oxide, with the balance being ignition loss. Magnesium aluminum silicates are commercially available from Vanderbilt under the tradename VEEGUM. A preferred grade of VEEGUM clay is sold under the tradename VEEGUM ULTRA. This grade has a pH of approximately 5 when in an aqueous  
20 dispersion, reducing the need to add pH buffering agents to the shampoo formula, and provides an attractive, light-colored shampoo. A 5% dispersion of this clay in water has a viscosity of 225-425 cps, using Vanderbilt Test Method 884. Preferably, the clay is present in an amount of from about 0.05 to 3.0, more preferably 0.10 to 1.0, % Active. If too much is included, a gel-like emulsion having undesirably high viscosity may result, if  
25 too little is included, the emulsion will tend to be unstable and the pigment may settle out of the shampoo.

In preferred shampoos, the glyceryl stearate and the clay are provided in a weight ratio of from about 3:1 to 10:1, more preferably about 5:1, and the pigment and clay are provided in a weight ratio of from about 1:3 to 1:8, more preferably 1:6. These  
30 ratios have been found to provide excellent product stability and resistance to phase separation as a result of temperature cycling, e.g., preferred shampoos are capable of withstanding at least 5 temperature cycles wherein the emulsion is held for 16 hours at 45°C, followed by 24 hours at 5°C, followed by 8 hours at 25°C, without visible phase separation, and more preferred shampoos are capable of withstanding 10 or more such  
35 temperature cycles.

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5                    Suitable pearl pigments are those pigments which will impart a pearlescent  
appearance to the shampoo formulation. Preferred pearl pigments include blends of mica  
and titanium dioxide, and titanium dioxide coated mica. Preferably, the ratio of mica to  
titanium dioxide in these preferred pigments is approximately 2:1, and the pigment has an  
average particle size of from about 2 to 85 micron. A particularly preferred titanium  
10 coated mica pigment is available from Rona, EM Industries, Inc. Chemicals and Pigments  
Division, Hawthorne, NY under the tradename TIMIRON MP-30. Preferably, the pearl  
pigment is included in an amount of from about 0.01 to 0.20, more preferably 0.025 to  
0.10, % Active. If too much is included, the composition will tend to be too pearled, and  
the pigment may tend to settle out of the shampoo matrix; if too little is included, the  
15 pearling effect will be reduced.

                  The compositions further include an amount of water sufficient to act as a  
carrier for the other components of the shampoo and to provide a desired viscosity in the  
final product. Preferably, the composition contains from about 30 to 45 percent water  
based on the total weight of the composition. It is also preferred that the water be purified  
20 or deionized.

                  Other additives may be included in the conditioning shampoo, including  
but not limited to fragrances, anti-dandruff agents, humectants, combing aids, processing  
aids, dyes, thickeners, proteins, herb and plant extracts, and other additives used in the  
formulation of shampoos.

25    Example 1

                  A shampoo having the ingredients shown in the table below was prepared  
by the following procedure:

1.                The purified water was charged to a mix tank and heated to 75°C. When  
the water reached this temperature, the smectite clay was added slowly and mixed until  
30 dispersed and uniform.
2.                The pearl pigment was then added and the mixture was mixed for about  
45-60 minutes at 75°C.
3.                While the temperature was held at 75°C, a premixed blend of ammonium  
lauryl sulfate, cocamidopropyl betaine, Lauramide DEA, and palmitic acid was added to  
35 the mixture and mixed for about 15 minutes.

- 5        4.            The DATEM was then added and the mixture was mixed for about  
20 minutes at 75°C until uniform.
5.            The insoluble silicone was then added and the mixture was mixed for about  
45-60 minutes at 75-80°C.
6.            The remaining cleansing agents were then added, reducing the temperature  
10 of the mixture to about 65°C.
7.            The mixture was then cooled to about 50°C.
8.            At 50°C the amino-functional silicone emulsion was added, and the  
mixture was mixed for about 15 minutes.
9.            The panthenol, tetrasodium EDTA, preservative, and humectant were  
15 added in that order, mixing after each addition until uniform.
10.           The batch was cooled to about 35°C and the wheat protein and fragrance  
were added in that order, mixing after each addition until uniform.

5	Ingredients	% Active	Purpose
	Ammonium laureth sulfate	9.667	Surfactant
	Ammonium lauryl sulfate	3.3330	Surfactant
	Sodium lauryl sulfate	4.0000	Surfactant
	Lauramide DEA	2.1750	Co-surfactant/foam booster
10	Cocamidopropyl betaine	1.2775	Surfactant
	Ammonium xylene sulfonate	0.5000	Surfactant/coupling agent
	Dimethicone	1.2500	Non-water soluble cond. agent
15	Trimethylsilylamodimethicone and octoxynol-40 and isolaureth-6	0.3000	Non-water soluble cond. Agent (water-soluble silicone emulsion)
	DATEM	1.5000	Emulsifier
	Palmitic acid	0.2750	Combing aid
	Glycerine	0.8000	Humectant
20	Magnesium aluminum silicate (VEEGUM ULTRA)	0.2500	Suspending agent
	Tetrasodium EDTA	0.1200	Chelating agent
	DMDM hydantoin and iodopropynyl butylcarbamate	0.0300	Preservative
	dl-Panthenol	0.0500	Body/shine enhancer
25	Hydrolyzed wheat protein and wheat oligosaccharides	0.0500	Body enhancer
	Mica and titanium dioxide	0.0500	Pearling agent
	Citric acid	1.0000	Processing aid (pH adjustment)
	Perfume 6041-AX	0.5000	Fragrance
30	Purified-water	q.s. to 100	Vehicle

5           This shampoo was tested on human hair by a number of shampoo users. These users reported that the shampoo provided good cleaning and conditioning, and left the hair feeling clean and conditioned, with little build-up observed even after daily use.

Other embodiment are within the claims.

For example, instead of, or in addition to, a pearl pigment, the shampoo  
10   may contain other types of pearling agents, e.g., liquid pearling agents such as glyceryl long chain esters, for example ethylene glycol distearate. If present, such pearling agents would be used in suitable quantities to impart the desired degree of pearling, as is well understood in the art. For example, if ethylene glycol distearate is used as the only  
15   pearling agent in the shampoo, preferred levels would generally be from about 0.10 to 0.25%.



CLAIMS

1. A conditioning shampoo comprising:  
a cleansing agent;  
an insoluble silicone conditioning agent;  
a stabilizing agent comprising a glyceryl ester and a smectite clay; and  
5 water.
2. The conditioning shampoo of claim 1, wherein said cleansing agent comprises ammonium laureth sulfate, ammonium lauryl sulfate, sodium lauryl sulfate, lauramide DEA, cocamidopropyl betaine, and ammonium xylene sulfonate.
3. The conditioning shampoo of claim 2, comprising from about 9 to 10%  
10 ammonium laureth sulfate, from about 3 to 4% ammonium lauryl sulfate, from about 3.5 to 4.5% sodium lauryl sulfate, from about 2 to 3% lauramide DEA, from about 1 to 2% cocamidopropyl betaine, and from about 0.25 to 0.75% ammonium xylene sulfonate.
4. The conditioning shampoo of claim 1, wherein said insoluble silicone conditioning agent comprises a mixture of a non-volatile polyalkyl siloxane and an amino-  
15 functional silicone derivative.
5. The conditioning shampoo of claim 4, wherein said polyalkyl siloxane comprises dimethicone and said amino-functional silicone derivative comprises trimethylsilylamodimethicone.
6. The conditioning shampoo of claim 2, wherein said insoluble silicone  
20 conditioning agent comprises a mixture of a non-volatile polyalkyl siloxane and an amino-functional silicone derivative.
7. The conditioning shampoo of claim 6, wherein said polyalkyl siloxane comprises dimethicone and said amino-functional silicone derivative comprises trimethylsilylamodimethicone.
- 25 8. The conditioning shampoo of claim 1, wherein said glyceryl ester comprises an ester of diacetyl tartaric acid glyceryl stearate.
9. The conditioning shampoo of claim 8, wherein said glyceryl ester is produced by reacting glycerol, stearic acid and diacetyl tartrate.
10. The conditioning shampoo of claim 9, wherein said glyceryl ester is  
30 DATEM.

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- 5      11.            The conditioning shampoo of claim 1, wherein said smectite clay comprises magnesium aluminum silicate.
12.            The conditioning shampoo of claim 10, wherein said smectite clay comprises magnesium aluminum silicate.
13.            The conditioning shampoo of claim 12, wherein said DA TEM and said  
10      magnesium aluminum silicate are provided in a weight ratio of from about 3:1 to 10:1.
14.            The conditioning shampoo of claim 1, further comprising a pearling pigment.
15.            The conditioning shampoo of claim 14, wherein said pearling pigment and said smectite clay agent are provided in a weight ratio of from about 1:3 to 1:8.
- 15      16.            The conditioning shampoo of claim 1, wherein said stabilizing agent is provided in an amount sufficient to enable the shampoo to exhibit no visible phase separation after 10 temperature cycles as defined hereinabove.
17.            A conditioning shampoo comprising:  
                 a cleansing agent comprising, based on the total weight of the shampoo,  
20      from about 9 to 10% ammonium laureth sulfate, from about 3 to 4% ammonium lauryl sulfate, from about 3.5 to 4.5% sodium lauryl sulfate, from about 2 to 3% lauramide DEA, from about 1 to 2% cocamidopropyl betaine, and from about 0.25 to 0.75% ammonium xylene sulfonate;  
                 an insoluble silicone conditioning agent;  
25                   a stabilizing agent; and  
                 water.
18.            A conditioning shampoo comprising:  
                 a cleansing agent;  
                 a conditioning agent;  
30                   a pearl pigment;  
                 a magnesium aluminum silicate; and  
                 water.
19.            A method of cleaning and conditioning hair comprising:  
                 (a) providing a shampoo comprising a cleansing agent, an insoluble  
35      silicone conditioning agent, a stabilizing agent comprising a glyceryl ester and a smectite

5 clay, and water;

(b) applying the shampoo to wet hair and massaging the hair to form a lather; and

(c) rinsing the lather from the hair.

20. A method of making a conditioning shampoo comprising:

10 (a) dispersing a smectite clay and a pearling pigment in water to form a pigment dispersion;

(b) adding one or more cleansing agents to the pigment dispersion;

(c) adding a glyceryl ester and an insoluble silicone to the pigment dispersion and mixing until a stable emulsion is formed; and

15 (d) adding an amino-functional silicone to the emulsion.

21. The method of claim 20, wherein step (a) is performed at about 70 to 80 °C.

22. The method of claim 20, wherein step (a) comprises pre-dispersing the smectite clay in the water, adding the pigment, and mixing the clay, pigment and water for about 40 to 70 minutes.

20 23. The method of claim 21, wherein step (a) comprises pre-dispersing the smectite clay in the water, adding the pigment, and mixing the clay, pigment and water for about 40 to 70 minutes.

24. The method of claim 20, wherein step (c) is performed at about 70 to 80 °C.

25 25. The method of claim 20, wherein step (c) comprises adding the glyceryl ester to the pigment dispersion, mixing until uniform, and then adding the insoluble silicone and mixing until emulsified.

26. The method of claim 24, wherein step (c) comprises adding the glyceryl ester to the pigment dispersion, mixing until uniform, and then adding the insoluble silicone and mixing until emulsified.

30 27. The method of claim 26, wherein after the addition of the insoluble silicone the mixture is mixed for about 40 to 70 minutes.

28. The method of claim 20, wherein steps (a) through (c) are performed at about 70 to 80 °C, and wherein the emulsion formed in step (c) is cooled to about 50 °C before the amino-functional silicone derivative is added.

# INTERNATIONAL SEARCH REPORT

Inter. Application No  
PCT/US 97/20620

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 A61K7/50 A61K7/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 368 187 A (M. FLOM ET AL.) 11 January 1983 see claim 1; example 1 ---	1, 14-19
X	DATABASE "CHEMICAL ABSTRACTS" (HOST: STN); Abs.120: 14 642, Columbus, OH, USA; & JP 05 246 824 A (SHISEIDO CO., Ltd) 24 September 1993 XP002059906 see the whole document ---	1
A	EP 0 673 639 A (L'OREAL) 27 September 1995 ---	1
A	GB 2 230 022 A (BP CHEMICALS LTD) 10 October 1990 see example D -----	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Name and mailing address of the ISA  
European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Glikman, J-F

# INTERNATIONAL SEARCH REPORT

information on patent family members

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